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Microwave pyrolysis of rice straw: Products, mechanism, and kinetics

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HIGHLIGHTS

- The highest energy densification ratio was achieved at the power level of 300 W.
- The atomic H/C and O/C ratios of rice straw were largely decreased.
- The primary components of gaseous product were CO, H_2, CO_2, and CH_4.
- More gaseous product was obtained at higher microwave power levels.
- Liquid production remained the same and showed a maximum of about 50 wt.%.

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ABSTRACT

Rice straw is an abundant resource for the production of biofuels and bio-based products. How to convert the recalcitrant lignocellulose effectually is a critical issue. The objective of this study was to investigate the products, mechanism, and kinetics of rice straw pyrolysis by using microwave heating. The highest energy densification ratio of solid residues was achieved at the microwave power level of 300 W. The atomic H/C and O/C ratios of solid residues were much lower than those of rice straw. The primary components of gaseous product were CO, H_2, CO_2, and CH_4, whose molecular fractions were 57%, 21%, 14%, and 8%, respectively. The more gaseous product and the less solid residues were obtained at higher microwave power levels, while the liquid production remained the same and showed a maximum of about 50 wt.%. The kinetic parameters of rice straw pyrolysis were increased with increasing microwave power level.

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1. Introduction

Biomass is a resource to produce renewable energy and fuels which are so-called bioenergy and biofuels. Biomass can be produced from various sources, such as wood, crops, agricultural and forestry residues, and biodegradable components of commercial, industrial, and municipal solid waste, which are some of the main renewable energy resources available (Bridgwater, 2006). The first-generation biofuels have caused some controversial issues such as food prices, deforestation, global warming and threats to biodiversity, because they come from the feedstocks of food crops (Luque et al., 2008). The second-generation biofuels, unlike the previous one, are generated from no longer food crops but rather non-food crops and biomass waste. Biomass waste means biological or biodegradable residues left from agricultural, forestry, industrial, and human activities. In the past, most of the biomass waste was not properly utilized or treated but directly burned or discarded on site. This old custom not only wastes the energy inside the biomass waste but causes CO_2 emission as well. The utilization of biomass waste can reduce the impact to the environment and produce useful fuels and chemicals. Besides, compared with other renewable resources giving heat and power, biomass represents the only source of liquid, solid, and gaseous fuels (Bridgwater and Peacocke, 2000).

Microwaves are electromagnetic waves with frequencies between 300 MHz and 300 GHz, and thus the corresponding wavelengths are between 1 mm and 1 m. Microwave heating includes two mechanisms: one is the dipole rotation, and the other is the ion migration. Both of them are able to heat materials quickly and uniformly. Microwave heating is also a selective and energy-saving technology without direct contacts with the heated materials (Jones et al., 2002). Therefore, it is widely used in many applications such as sample pretreatment (Roig, 1995), synthesis (de Andresa et al., 1999), digestion (Bettinelli et al., 2000), extraction (Perez Cid et al., 2001), and sludge stabilization (Chen et al., 2005; Hsieh et al., 2007). The technology of pyrolysis induced by microwave heating has been researched to treat various feedstocks such as oil shale (El harfi et al., 2000), oil-palm stone (Guo and Lua, 2000), paper (Miura et al., 2001), plastic waste (Ludlow-Palafox

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and Chase, 2001), sewage sludge (Menendez et al., 2002), rock phosphate (Bilali et al., 2005), scrap tire (Appleton et al., 2005), coffee hulls (Menendez et al., 2007), wood (Chen et al., 2008), rice straw (Huang et al., 2008), corn stalk bale (Zhao et al., 2010), oil palm biomass (Salema and Ani, 2011), and microalgae (Hu et al., 2012). These researches have shown that, by using the microwave pyrolysis, biomass feedstocks can be converted into various products such as bio-oil, bio-char, fuel gas, and hydrogen. The reaction performance and productivity depends on factors including operating parameters, biomass characteristics, reaction atmosphere, and catalytic effect.

Rice is one of the most important food crops, and its annual worldwide production has been over 700 million metric tons since 2010 (Food and Agricultural Organization of the United Nations (FAO), 2012). Therefore, close to 1 billion metric tons of rice straw can be generated annually, because about 1.35 metric tons of rice straw remains in the field for every metric ton of grain harvested (Kadam et al., 2000). This is really a great quantity of biomass waste or the source of biofuels and bio-based products depending on how to deal with the rice straw. The biochemical treatment of lignocellulosic biomass is difficult due to the natural resistance of plant cell walls to microbial and enzymatic deconstruction (known as biomass recalcitrance), which is largely responsible for the high cost of lignocellulose conversion (Himmel et al., 2007). Thermochemical treatments including pyrolysis and gasification can effectually overcome the biomass recalcitrance by decomposing the crystalline recalcitrant structure of lignocellulose. In order to establish a feasible technology for the treatment of biomass waste like rice straw, this study aims at investigating the products, mechanism, and kinetics of rice straw pyrolysis by using the microwave heating.

2. Methods

2.1. Material

The sample of this study was rice straw provided by Industrial Technology Research Institute (ITRI), Taiwan. The rice straw was stored at room temperature for months to maintain constant moisture content, and then it was shredded and sieved by a 50-mesh (0.297 mm) screen. The basic components and properties of rice straw have been reported in the previous article (Huang et al., 2012). The combustible and ash contents (dry basis) of rice straw were about 90 and 10 wt.%, respectively. The hemicellulose, cellulose, and lignin contents (dry basis) of rice straw were about 20, 42, and 26 wt.%, respectively. The C, H, and O contents (dry and ash-free basis) of rice straw were about 46, 6, and 46 wt.%, respectively. The higher heating value (HHV) of rice straw was about 16 MJ/kg.

2.2. Experimental device and procedure

This study used a single-mode (focused) microwave device with 2.45 GHz frequency. The schematic diagram of overall microwave pyrolysis set-up can be found elsewhere (Wang et al., 2010). The shredded and sieved biomass feedstock was filled in a quartz crucible. Then it was placed inside a quartz tube and precisely in the pathway of microwaves. In order to maintain anoxic circumstances, Nitrogen gas was purged into the system with a flow rate of 50 mL/min. After enough purging to maintain an inert atmosphere, the power supply was turned on and switched to the prescribed microwave power level for the prescribed processing time. Reflection microwave power levels were controlled to be as low as possible during the whole experiment period. When the prescribed processing time was reached, the power supply was turned off and the carrier gas purging was stopped, and then the tar and gas collectors were removed and sealed. After self-cooled down to about 100 °C, solid residues were removed and placed in a desiccator for hours. All the experiments were repeated twice to obtain the results in average values.

2.3. Product analysis

The proximate analysis referred to the standard test method D5142 of American Society for Testing and Materials (ASTM). The elemental analysis was carried out by a Perkin–Elmer 2400 Elemental Analyzer. The heating values of raw rice straw and solid residues were determined by a CAL2K ECO calorimeter. The analysis of gaseous product was carried out by a Perkin–Elmer Auto System XL gas chromatography–thermal conductivity detector (GC–TCD) with a Supelco Carboxen 1010 PLOT column. The temperatures of injector, oven, and detector were 120, 100, and 150 °C, respectively. The flow rate of carrier gas (He/N_2) was 10 mL/min (25:1 split). The liquid product was analyzed by a Perkin–Elmer Turbo Mass Gold gas chromatography–mass spectrometry (GC–MS) with a Supelco Equity–5 capillary column. The initial temperature of oven was 45 °C and held for 3 min, and then it was ramped from 45 °C to 300 °C at the rate of 5 °C/min and held for 5 min. The flow rate of carrier gas (He) was 10 mL/min (20:1 split).

3. Results and discussion

3.1. Performance of microwave heating

The rice straw sample was efficiently heated by the microwave radiation. The temperature was not obviously increased at the first minute, and most of the heating effect occurred at about 1–10 min, as reported in the previous article (Huang et al., 2012). This should be owing to that the rice straw did not absorb enough microwaves yet at the first minute. Once the microwave energy was sufficient, the temperature was effectually increased. The maximum temperature and the maximum heating rate were governed by how much the input energy is (i.e., the microwave power level). The maximum temperature and the maximum heating rate were both increased when increasing the microwave power level, as shown in Fig. 1. The correlation between the maximum temperature and the microwave power level was linear, while the maximum heating rate exponentially increased with increasing microwave power level. Therefore, the maximum heating rate was more sensitive to the microwave power level than the maximum temperature. This means that at higher microwave power levels, the maximum temperatures can be reached more quickly, and thus the processing time can be shorter. Generally, it only took 5–10 min to reach the maximum temperatures at higher microwave power levels (400–500 W), while the temperatures kept climbing very slowly

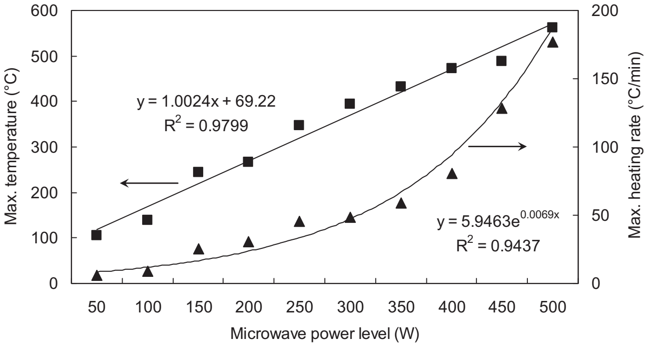

Fig. 1. The maximum temperatures and the maximum heating rates at different microwave power levels.

till the end of the experiment at lower microwave power levels (50–250 W). Thus, for the performance of microwave heating, there would be a critical microwave power level which was affected by some operational factors such as reactor type and sample size. Less than the critical value would lead to low heating performance. In this study, the critical microwave power level should be 400 W.

3.2. Solid residues

After microwave pyrolysis, the HHV of solid residues was higher than that of the raw rice straw due to the reduction of low-HHV hydrocarbons (e.g., hemicellulose). Table 1 lists the HHV and the mass yield of solid residues at different microwave power levels. The mass yield means the ratio of solid residues mass to biomass sample mass:

$$\text{Mass yield} = \frac{\text{Mass of solid residues}}{\text{Mass of biomass sample}} \times 100\% \qquad (1)$$

It can be seen that the mass yield was lower at higher microwave power level, which represents that the more rice straw was converted into liquid and gaseous products. The highest HHV of solid residues occurred at the middle microwave power level (300 W). This should be owing to that part of the fixed carbon content was removed when the microwave power level was too high (Huang et al., 2008). To further evaluate the energy preserved in the solid residues after microwave pyrolysis, this study applies the measures of energy densification ratio (EDR) and energy yield defined as follows (Yan et al., 2009):

$$\text{Energy densification ratio} = \frac{\text{HHV of solid residues}}{\text{HHV of biomass sample}} \qquad (2)$$

$$\text{Energy yield} = \text{Mass yield} \times \text{Energy densification ratio} \qquad (3)$$

The energy yields and the EDR at different microwave power levels are shown in Fig. 2. The EDR increased with increasing microwave power level to meet a maximum of about 1.3 at 300 W, and then it decreased with further microwave power level. At the microwave power levels of 400–500 W, the EDR was only 1.05–1.1, which means that the HHV of solid residues was only a bit higher than that of raw rice straw. Compared with the EDR, the energy yield can present the proportion of overall energy of solid residues to biomass sample more precisely. The energy yield decreased with increasing microwave power level and did not show a maximum like the EDR. The energy yield was about 65% at the microwave power level of 150 W, while it was only about 20% at 500 W. Therefore, if the solid residues of high energy yield and EDR are preferred, the microwave power level should not exceed 300 W. This mild pyrolysis is commonly named as torrefaction. On the other hand, if the highest conversion of rice straw into liquid and gaseous products is expected, the microwave power level should be as high as possible. The production of liquid and gaseous products will be discussed later.

Table 1
The mass yield and higher heating value (HHV) of solid residues.

Microwave power level (W)	Mass yield (%)	HHV (MJ/kg)
Raw rice straw	–	16.16
150	54.65	19.07
200	39.59	19.66
250	34.87	19.84
300	28.07	20.94
350	25.29	19.31
400	24.54	17.64
450	23.08	17.44
500	19.62	16.88

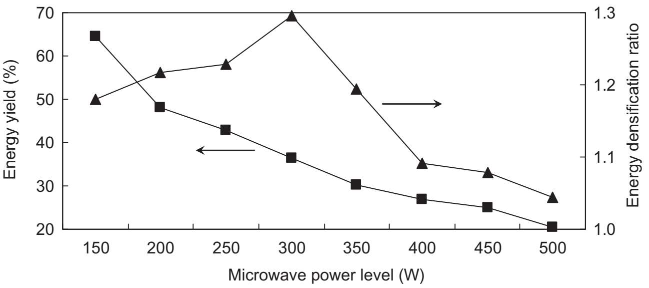

Fig. 2. The energy yields and the energy densification ratios of solid residues at different microwave power levels.

The van Krevelen diagram, which is a plot of atomic H/C ratios versus atomic O/C ratios, is shown in Fig. 3. The atomic H/C and O/C ratios of rice straw were much higher than those of coals. After microwave pyrolysis, both ratios decreased with increasing microwave power level. At the microwave power levels of 400–500 W, the ratios of solid residues were close to those of lignite and bituminous coal. Tumuluru et al. (2011) pointed out that less atomic pointed out that less atomic H/C and O/C ratios may contribute to less smoke and water–vapor formation and less energy loss during combustion and gasification processes. Therefore, the microwave power level should be higher when considering the thermal treatment of solid residues. However, this would be against the conclusion from the viewpoint of energy yield.

3.3. Gaseous product

The gaseous product means the incondensable part of the vapor generated from the microwave pyrolysis of rice straw. The molar quantities of major components (H_2, CH_4, CO, and CO_2) in the gaseous products at different microwave power levels are shown in Fig. 4. All the productions of the components generally increased with increasing microwave power level linearly. By comparing Figs. 1 and 4, it can be seen that more gases can be obtained at higher reaction temperature. The overall molecules of the four gases were about 12 mmol at 150 W, which was promoted to be about 73 mmol at 500 W. The microwave power level did not much affect the fractions of the gases. The average percentages of H_2, CH_4, CO, and CO_2 were 21, 8, 57, and 14%, respectively. The high H2 content of gaseous product is coincident with the works of Menendez et al. (2007), Fernandez et al. (2009). More specifically, from 150 W to 500 W, the H_2 fraction increased by 4% but the CH_4 fraction decreased by 4%. This may imply that the steam

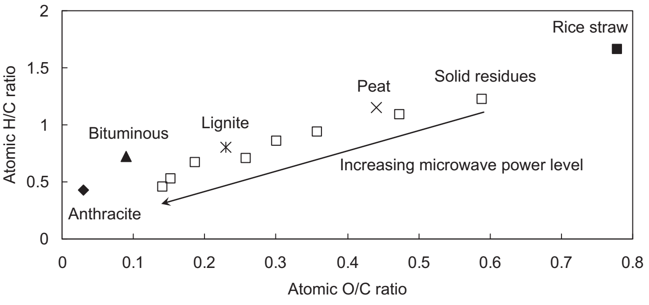

Fig. 3. The van Krevelen diagram for coals, rice straw, and solid residues at different microwave power levels. The data of coals are taken from Ullmann (2003).

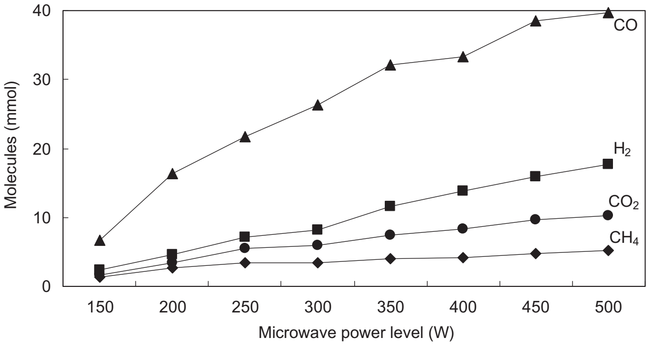

Fig. 4. The compositions of gaseous products at different microwave power levels.

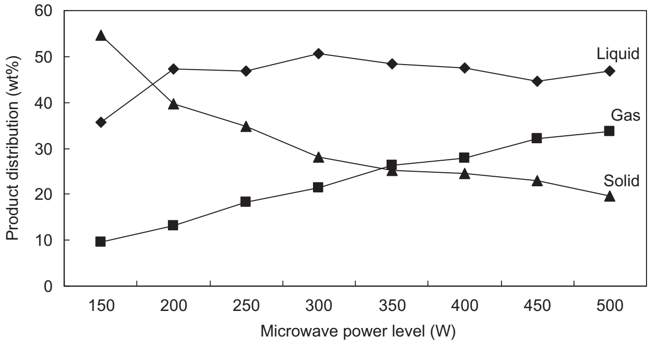

Fig. 5. The product distributions at different microwave power levels.

reforming of methane is remarkable at higher microwave power levels:

$$CH_4 + H_2O \leftrightarrow CO + 3H_2 \tag{4}$$

The CO_2 fraction did not change with the microwave power level obviously. At the microwave power levels of 200–300 W, the CO fraction was relatively higher while the H_2 fraction was relatively lower. This could be owing to the water–gas shift reaction:

$$CO_4 + H_2O \leftrightarrow CO_2 + H_2 \tag{5}$$

Furthermore, when the microwave power level was high enough (>400 W), the fractions of H_2, CH_4, CO, and CO_2 were 24%, 7%, 55%, and 14%, respectively, and did not change anymore. This may imply that the overall effect of the complicated interactions between gases would become stable at the higher microwave power levels.

3.4. Product distribution

In addition to solid residues and gaseous product, there was also a liquid product collected by the condensation of product vapor. The solid residues and gaseous product can be quantified but the liquid product cannot. Part of the vapor condensed on the tube wall, so it was difficult to collect the liquid product completely. Therefore, the quantity of liquid product was determined by difference. The liquid product was mainly composed of monoaromatics (benzene, phenol, and their alkyl derivatives), aliphatics (alkanes and alkenes), and polycyclic aromatic hydrocarbons (PAHs). The product distributions at different microwave power levels are shown in Fig. 5. Generally, more gaseous product and less solid residues were obtained at higher microwave power level. The fractions of solid residues and gaseous were about 50 and 10 wt.% at 150 W and were about 20 and 34 wt.% at 500 W, respectively. However, the liquid fractions were almost the same (about 50 wt.%) for all the microwave power levels except 150 W. Therefore, there should be a maximum for the liquid production.

3.5. Reaction mechanism

According to the product distributions and liquid yields as above mentioned, a possible reaction mechanism of microwave pyrolysis of rice straw as shown in Fig. 6 was proposed by referring to the Broido–Shafizadeh model (Bradbury et al., 1979):

$$\text{Cellulose} \rightarrow \text{Active cellulose} \begin{array}{l} \rightarrow \text{Char} + \text{Gases} \\ \rightarrow \text{Volatile tars} \end{array} \tag{6}$$

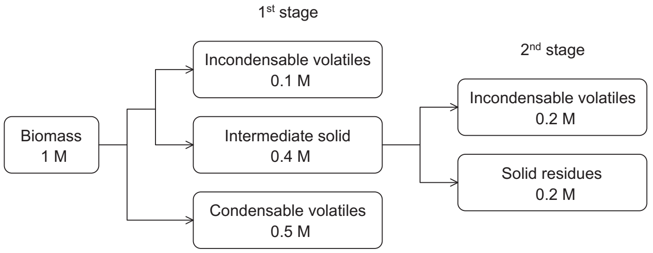

Fig. 6. The mechanism and mass balance of microwave pyrolysis of rice straw (M: mass unit).

Since the liquid yields at the microwave power levels of 200–500 W were almost the same, the further cracking and reforming of liquid product as reported by Ahmed and Gupta (2009) could be neglected. At the microwave power level as low as 200 W, the gasification (2nd stage) seldom happened, so 1 M (mass unit) rice straw was pyrolyzed to produce 0.5 M liquid, 0.4 M solid, and 0.1 M gas. When the microwave power levels were higher, the intermediate solid would undergo gasification to produce more gases. At 500 W, about half the intermediate solid was converted into gases, so the final yields of liquid, solid, gas were 0.5, 0.2, and 0.3, respectively. Further gasification of solid residues should be difficult, because the solid yield was close to the sum of fixed carbon and ash contents of rice straw.

One of the important features of the Broido–Shafizadeh model is a temperature dependent competition between two reactions determining the ratio of tar to gases plus char (Reynolds and Burnham, 1997). However, the ratio of tar to gases plus char did not change a lot, since the liquid yields at 200–500 W were almost the same. This may imply that, once the temperature is higher than a certain extent (e.g., the maximum temperature at 200 W: 266 °C), the liquid production would be limited or even stopped. The limited conversion from biomass to liquids may be owing to the lignocellulosic composition of biomass. The thermo-chemical behaviors of lignocellulosic contents (i.e., cellulose, hemicellulose, and lignin) are different from one another. Therefore, different biomass feedstocks with different lignocellulosic contents may have different maximum liquid yields.

3.6. Kinetic analysis

Due to the difficulty in acquiring sufficient thermogravimetric data of microwave pyrolysis of rice straw, an isoconversional method was used in an attempt to carry out the kinetic analysis. The experiment of microwave pyrolysis cannot provide sufficient data as the thermogravimetric analysis (TGA) can due to the experimental setup. Therefore, this study tried to carry out the kinetic analysis by assuming a pseudo first-order model for the microwave pyrolysis of rice straw. The rate equation for the first-order reaction can be expressed as:

$$d\alpha/dT = (A/\beta)\exp(-E/RT)(1-\alpha) \tag{7}$$

Table 2
The kinetic parameters of microwave pyrolysis of rice straw.

Conversion fraction	E (kJ/mol)	A (1/s)	R^2
0.72	21.84	0.34	0.94
0.76	27.13	1.06	0.97
0.80	29.88	1.60	0.90

where T is the temperature, β is the heating rate, A is the pre-exponential factor, E is the activation energy, R is the universal gas constant, and α is the conversion fraction of sample:

$$\alpha = (m_0 - m_t)/(m_0 - m_f) \tag{8}$$

where m_0 is the initial mass of biomass sample, m_t is the mass at any time, and m_f is the final mass at the end of reaction. After approximate integration and taking natural logarithms, Eq. (6) becomes:

$$\ln\beta = (-E/R)(1/T) + \ln(-ART^2/E/\ln(1-\alpha)) \tag{9}$$

After plotting $\ln\beta$ versus $1/T$, the slope and intercept obtained by linear regression were used to determine the activation energy and pre-exponential factors at different conversion fractions, as listed in Table 2. The average conversion fractions of 0.72, 0.76, and 0.80 were carried out at the microwave power levels of 200–300, 300–400, and 400–500 W, respectively. Due to the complexity of microwave pyrolysis, the coefficients of determination (R^2) were lower than those of conventional thermogravimetric analyses.

The activation energy and pre-exponential factor were both higher at higher conversion fraction which was carried out by higher microwave power level. At the conversion fractions of 0.72, 0.76 and 0.80, the activation energy was 21.84, 27.13 and 29.88 kJ/mol, and the pre-exponential factors were 0.34, 1.06 and 1.60 1/s, respectively. Besides, all of these kinetic parameters were much higher than those of microwave torrefaction which were reported in the previous article (Huang et al., 2012). Therefore, the more biomass conversion would need the more energy input to overcome the energy barrier of thermal-resistant biomass components and to provide higher probabilities of molecular reactions. Consequently, the reaction rate was increased with increasing microwave power level. The kinetic parameters obtained here should be useful for further researches of biomass pyrolysis by using the microwave heating. The future works may include the adjustment of operating condition, the scaling-up and optimization of reaction system, and the maximum production of biofuels and bio-based materials.

4. Conclusion

The calorific value of solid residues was highest at the microwave power level of 300 W. The atomic H/C and O/C ratios of solid residues were decreased with increasing microwave power level. The primary components of gaseous product were CO, H_2, CO_2, and CH_4, and the liquid product was mainly composed of monoaromatics, aliphatics, and PAHs. The more gaseous product and the less solid residues were obtained at higher microwave power levels, while the liquid production remained the same and showed a maximum of about 50 wt.%. Therefore, the further cracking and reforming of liquid product could be neglected.

Acknowledgements

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